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Subject: MLI Implementation Project Report

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Objective

Due to the divestiture of ARCO Metals, the proposal for the MLI Implementation presented in Columbia Falls, Montana in April of 1984 was revised. The revised plan was to make six anodes to provide a representative mass, and bake them to allow final adjustments on the furnace. There was no provision for verifying correct operation of the furnace.

Summary

The test work consisted of making six anodes following the screen curve used for the MLI test anode made in Columbia Falls, Montana in June of 1984. The anodes were baked and the following tests performed, Baked Apparent Density (BAD), Electrical Resistivity (ER), and Compressive Strength (CS).

The anodes were made using old materials on hand to provide a fairly representative mass to debug the furnace operation. During the adjustment and debugging of the furnace and controller, the paste was heated to 100°C several times. The results from these anodes are average BAD of 1.383 g/cc, average ER of 0.0030 ohm-in, and average CS of 3054 psi. These results are very poor, and cannot be taken as an indication of the performance of the furnace since this run was intended only for debugging.

It is recommended to bake another set of anodes using the parameters in Table III to allow final debugging and verification of the furnace operation.

Procedure

Collier coke and Reilly pitch, both obtained in 1982, were used to make paste. The pitch was received in flakes, and the drum was roughly 1/3 full which undoubtedly caused oxidation of the pitch. The coke was screened using an eighteen inch Sweco screen down to 100 mesh. The finer coke was produced by roll crushing and ball milling. Coke minus 200 m was screened using an ATM sonic sifter, Model P-60.

The aggregate distribution used was based on the MLI test anode screen curve used at Columbia Falls, Montana in June of 1984. The distribution of +0.371 in and -200 m material was based on MLI screen curves received in March of 1984. The distribution used is shown below in Table I.

TABLE I - Aggregate Distribution Used in Tucson

Mesh Size	% Wt
-0.525 + 0.371"	1.9
+4 m	19.1
+8 m	25.4
+14 m	12.2
+28 m	3.6
+48 m	2.2
+100 m	3.3
+200 m	10.9
+325 m	9.0
-325 m	12.4

The pitching level used was 25.4%, the MLI normal paste composition.

The paste was mixed in 7.5 kg batches in our sigma-blade mixer at an average mixing temperature of 165°C. The data sheets show a mixing temperature of 170°C as measured with a type T thermocouple. Test work done for Sebree showed that the type T thermocouple read about 5°C higher than the type K thermocouple. The total mixing time was 30 minutes, with temperature readings taken at 15 minute intervals. The mixer was turned off at 15 minutes to scrape paste from the mixer sides and blades.

Paste was taken from the mixer and put directly into the sample tubes. After about 2 large scoops had been transferred, the paste was pounded vigorously for about 10-15 strokes using the long handled pounding tool. For the initial mix, we put the paste into a metal tray before putting it in the sample tube. We stopped this because the paste cooled too rapidly and became unworkable. We found that three and one half batches filled a sample tube. This left the top baking weight sticking out of the tube by 1 to 2 inches. The tubes were marked one through six using magic marker. The marker remained legible after baking.

After all the mixes were made, the anodes were loaded into the furnace. The lid and two pieces of M-Board were put in place first. We cut a rectangular opening in the M-Board for the anodes, and put the steel lid in place. We used the gantry crane to load the anodes. We placed a piece of $\frac{1}{4}$ inch rubber around the tube under the wire sling to keep the sling from slipping.

After the anodes were in place, we put more M-Board on top of the steel lid. We put the heating blankets around the tubes. The rivets were put on the wrong sides, so the springs supplied were too short. However, 2 springs latched together fasten the top and bottom. Two pieces of baling wire held the blankets quite well. The wire was tightened to allow a thermocouple to fit between blanket and tube.

Hooked three TCs up to a digital readout, and moved the TCs around to check the temperature of each upper sample tube. Six TCs would be ideal, but we were short on TCs. Each blanket comes with a built in thermostat. Determined that a setting about halfway between medium and high kept the temperature at 115°C. Plug heating blankets into the six outlets mounted on the furnace. They are wired for 220 V, but they will also run on 110 V. Before plugging the blankets in, turn the thermostats to low and leave them there for a few minutes before increasing the settings. The tubes crackle and smoke if the blankets heat up too rapidly.

When the blankets were in place, packed loose Cerafiber around the tubes to fill the gap between tube and lid. The steel restraining frame was put over the anodes last. Used C-clamps to fasten the frame to the furnace. The frame fits very snugly against the lid.

We tuned the controller for the furnace after a representative mass was in the furnace. This is a trial and error process, and the furnace heated up to 100 degrees several times, and overshot to 250 degrees once. A set of constants was arrived at and are listed below in Table II.

TABLE II - Initial Tuning Constants

Output limit	30
Proportional band	4
Reset	0.1
Rate	0.1
Lineout	10

The furnace heated up at the rate specified by MLI. However, as pitch volatiles were evolved and hoods were turned on, the internal temperature stayed about 10 to 15 degrees below the setpoint. This remained constant up to 550°C.

To eliminate this problem, I would try the following tuning constants, shown in Table III.

TABLE III - Recommended Tuning Constants

Output limit	40
Proportional band	2
Reset	0.1
Rate	0.1
Lineout	5

By increasing the amount of power to the furnace and decreasing (narrowing) the proportional band, should be able to maintain the setpoint temperature, and heat up at the required rate.

Once the furnace reached 200°C, the heating blankets were turned on to about 115°C. The blankets were turned off at 400°C. The MLI lab manual does not clearly state that the upper portion should stay at 110°C until the bake cycle is finished. With the exhaust system we used, the blankets should have been left on for the entire bake cycle to maintain the desired temperature. With a different exhaust system, the blankets could possibly be turned off.

After the bake cycle was complete, the furnace was turned off. Once the furnace cooled to about 300°C, removed the insulation from the lid, removed the Cerafiber, and took the heating blankets off. When the furnace was completely cool, removed the lid and anodes. Took the baking weights out first. Two weights were stuck and wouldn't come out. Took them out forcibly after the sample had been cut off. Noticed a layer of black material about 1" thick on the bottom of the furnace liner, appears to be anode material.

Cut off the bottom 300 mm of anode using the horizontal bandsaw with a carbide blade. The oil pump was turned off. Gave a good, straight cut, but took about 25 minutes per cut. The bottom section was thrown out, as was the top portion once the 250 mm sample was obtained. Used a Dremel with an abrasive 1" cut-off wheel to cut the stainless sample tube. Took about 10 minutes and five wheels per anode. Worked very well, must be slow and careful to avoid breaking wheels.

Used a vertical bandsaw to cut 3 anodes lengthwise. Used a remington sawblade, which worked fairly well until blade became dull. Got very poor cuts after that, due to the sawblade bowing while cutting. A diamond saw would work much better here, want one where the blade height is constant and the sample moves. Our diamond saws would not take the sample length, so I used the bandsaw.

After the samples were quartered, I machined three pieces to about 2.25 inches in diameter. I purposely left them larger than I needed, in case they shrank during baking or the fluid coke stuck to the surfaces. Our lathe has a three jawed chuck which worked quite well. Finding the center to get the correct diameter is the tricky part, the actual machining is no problem as long as small cuts are taken initially.

After the samples were machined and labelled (I used chalk which remains intact through baking), they were loaded into a steel muffle. The 9 anodes were placed upright with about 1½ inches between samples, and fluid coke added to about 2 inches above the samples. Some Cerafiber was placed on top of the fluid coke to fill the space between the coke and muffle lid. The anodes were baked according to MLI's second cycle bake rate. The anodes were allowed to cool to about 100°C before they were removed from the furnace.

The anodes were machined to the final 2 inch diameter after cooling to room temperature, and cut in half lengthwise. The diameter did not change noticeably during the second bake. Fluid coke was trapped in

the surface voids, but there was no adhering. On samples that are not as poor as these, there should be no entrapment of fluid coke. The anodes can be machined to the final diameter before the final bake.

Baked apparent density (BAD) and electrical resistivity (ER) were run on samples 2 inches in diameter and about 5 inches long. The procedures given in report number 83-TP-5, "Columbia Falls Anode Optimization, Phase II," Appendix A were used for BAD, ER and compressive strength (CS). For the CS, 5 inch samples were cut in half lengthwise in order to give duplicate results and have samples intact to send to Columbia Falls, Montana.

Results

Results for BAD, ER, and CS are shown in Table IV. Anodes 6-1 and 6-2 only have one sample because part of each anode was ruined during cutting.

TABLE IV MLI IMPLEMENTATION DATA

Anode Number	Baked Apparent Density (g/cc)	Electrical Resistivity (ohm-cm)	Electrical Resistivity (ohm-in)	Compressive Strength (psi)
4-1-A	1.413	0.0074	0.0029	3381
				3386
4-1-B	1.416	0.0070	0.0028	
4-2-A	1.412	0.0070	0.0028	3741
				2759
4-2-B	1.409	0.0071	0.0028	
4-3-A	1.395	0.0072	0.0028	3274
				3270
4-3-B	1.404	0.0070	0.0028	
5-1-A	1.348	0.0081	0.0032	
5-1-B	1.359	0.0076	0.0032	
5-2-A	1.362	0.0077	0.0030	2562
				2562
5-2-B	1.366	0.0079	0.0031	
5-3-A	1.373	0.0074	0.0029	2991
				2836
5-3-B	1.370	0.0076	0.0030	
6-1	1.389	0.0075	0.0029	3611
6-2	1.372	0.0078	0.0031	
6-3-A	1.361	0.0077	0.0030	2954
				2356
6-3-B	1.374	0.0075	0.0030	3076

Averages and standard deviations for the values are shown in Table V.

TABLE V Means and Standard Deviations of the Anodes Tested

Anode Number	BAD (g/cc)		ER (ohm-cm)		ER (ohm-in)		CS (psi)	
	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev
4	1.408	0.007	0.0071	0.0001	0.0028	0.00004	3302	289
5	1.363	0.008	0.0077	0.0002	0.0031	0.0001	2738	184
6	1.374	0.010	0.0076	0.0001	0.0030	0.0001	2999	446

The anodes are very porous, and the BAD and CS values shown are correspondingly low. The low BAD values can be attributed to the following three conditions:

(1) the age of the pitch,
(2) the repeated heating and cooling of the paste while tuning the controller, and
(3) the material in the bottom of the furnace muffle, which appeared to be fines and pitch from the anodes.

These anodes were made strictly to provide a representative mass to heat in the furnace while debugging furnace operation.

Recommendations

- Bake some paste using the tuning constants recommended in Table III to avoid the difference between the setpoint and actual temperatures.
- Use a diamond saw to cut the anodes into lengthwise sections.
- Keep track of where the anode sections were in the furnace to check for temperature variations within the furnace.

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I. VOGELMANN

IV:dg

cc: D. F. Ryan/CF
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kw: 5.1.2
Carbon
Anodes
MLI